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HAIR COSMETIC MATERIAL

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## Abstract

### Constitution

A hair cosmetic material, characterized by the fact that it contains component (A) and component (B) in the following:

- (A) Microparticles of a fluorine-containing olefin resin
- (B) One or more silicones selected from a group consisting of dimethyl polysiloxane, methyl phenyl polysiloxane, amino-modified silicone, fatty acid-modified polysiloxane, alcohol-modified silicone, aliphatic alcohol-modified polysiloxane, polyester-modified silicone, epoxy-modified silicone, fluorine-modified silicone, cyclic silicone, and alkyl-modified silicone.

### Effect

The adherence of dirt can be prevented, the hair-setting maintaining power is strong, and a good feel to the touch is rendered.

## Claims

1. A hair cosmetic material, characterized by the fact that it contains component (A) and component (B) in the following:

- (A) microparticles of a fluorine-containing olefin resin
- (B) one or more silicones selected from a group consisting of dimethyl polysiloxane, methyl phenyl polysiloxane, amino-modified silicone, fatty acid-modified polysiloxane, alcohol-modified silicone, aliphatic alcohol-modified polysiloxane, polyester-modified silicone, epoxy-modified silicone, fluorine-modified silicone, cyclic silicone, and alkyl-modified silicone.

2. The hair cosmetic material described in Claim 1, in which the microparticles of a fluorine-containing olefin resin of component (A) are polyfluoroethylene (PTFE) and/or a tetrafluoroethylene-hexafluoropropylene copolymer (FEP).

3. The hair cosmetic material described in Claim 1, in which the maximum diameter for the microparticles of a fluorine-containing olefin resin of component (A) is 30  $\mu\text{m}$  or less and the average particle diameter is 20  $\mu\text{m}$  or less.

4. The hair cosmetic material described in Claim 1, in which the content of the microparticles of a fluorine-containing olefin resin of component (A) is 0.01–50 wt %.

## Detailed explanation of the invention

[0001]

### Industrial application field

The present invention relates to a hair cosmetic material that prevents the adherence of scalp oils, other oily dirt, dust, and so on, that is excellent in the style maintaining capability for

hair in rain or at high humidity, and that gives a smooth feel to hair, by maintaining water-repelling and oil-repelling characteristics.

[0002]

Prior art

With an increase in hair-washing frequency, the number of persons complaining about damage to the hair and scalp due to excessive washing has increased. The inhibition of damage to hair and so on has been carried out with hair rinses, conditioners, treatments, etc.

[0003]

However, the current situation is that the prevention of the adherence of dirt to hair due to the inhibition of excessive washing has not been investigated to a large extent.

[0004]

As a method for the prevention of adherence of dirt to hair, maintenance of oil-repelling characteristics on the hair surface can be considered. For example, a method for the treatment of hair with perfluoropolyether (such as Japanese Kokai Patent Application No. Hei 2[1990]-174711 and so on) or silicone can be mentioned. The hair treated with perfluoropolyether exhibits good dirt-preventing characteristics. However, said perfluoropolyether has a high cost. In addition to its feel to the difficulty in stable blending into a product, its feel to the touch is also inferior to silicone. On the other hand, silicone shows water-repelling characteristics. It has already been used in waterproof sprays and so on. However, durability is poor. Furthermore, since silicone itself is a soft polymer with a small intermolecular force, it has disadvantages in which not only is there no hair-setting maintaining power, but also the hair-setting power is decreased owing to other components.

[0005]

Problems to be solved by the invention

Therefore, the objective of the present invention is to provide a hair cosmetic material that can prevent the adherence of dirt to hair, that has a strong hair-setting maintaining power, and that renders a good feel to the touch.

[0006]

Means to solve the problems

As a result of carrying out zealous investigations in view of such an actual situation, the present inventors have discovered that a hair cosmetic material containing microparticles of a

fluorine-containing olefin resin and silicone gives water-repelling and oil-repelling characteristics, renders a dirt-preventing effect, and renders a style maintaining capability even under high humidity, regardless of whether it contains silicone. By kneading a fluorine-containing olefin resin in a silicone beforehand, the fluorine-containing olefin resin is carried strongly. Therefore, it does not flow even by washing with water. Furthermore, the hair can be finished in a smooth manner with luster. The present invention has been accomplished.

[0007]

In other words, the present invention provides a hair cosmetic material, characterized by the fact that it contains component (A) and component (B) in the following:

(A) Microparticles of a fluorine-containing olefin resin,

(B) One or more silicones selected from a group consisting of dimethyl polysiloxane, methyl phenyl polysiloxane, amino-modified silicone, fatty acid-modified polysiloxane, alcohol-modified silicone, aliphatic alcohol-modified polysiloxane, polyester-modified silicone, epoxy-modified silicone, fluorine-modified silicone, cyclic silicone, and alkyl-modified silicone.

[0008]

As resins of microparticles of the fluorine-containing olefin resin as component (A) for use in the present invention, for example, polytetrafluoroethylene (PTFE), polyhexafluoropropylene, polychlorotrifluoroethylene, a tetrafluoroethylene-hexafluoroethylene copolymer, a tetrafluoroethylene-hexafluoropropylene copolymer (FEP) and so on can be mentioned. Among these, the polytetrafluoroethylene (PTFE) and the tetrafluoroethylene-hexafluoropropylene copolymer (FEP) are preferred.

[0009]

It is preferable that these microparticles of the fluorine-containing olefin resin have a maximum particle diameter of 30  $\mu\text{m}$  or less, especially 5  $\mu\text{m}$  or less, from the aspects of smoothness, softness, agent appearance, stability, etc. Furthermore, it is preferable that the average particle diameter is 20  $\mu\text{m}$  or less, especially 3  $\mu\text{m}$  or less. It is possible that one or more of these microparticles of the fluorine-containing olefin resin can be used in combination. If they are blended at 0.01–50 wt%, especially 1–20 wt%, in the total composition, it will be desirable from the aspects of oil-repelling characteristics, smoothness, softness, agent appearance, stability, etc.

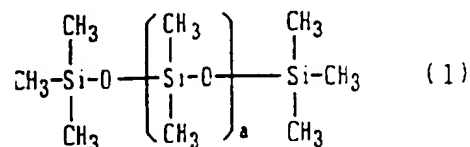
[0010]

Furthermore, as silicones of component (B) that can be used in the present invention, for example, (B-1)-(B-11) shown in the following can be mentioned.

(B-1) The dimethyl polysiloxane represented by the following formula (1)

[0011]

[Structure 1]



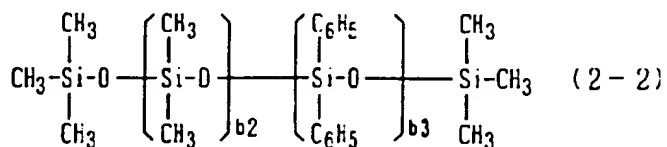
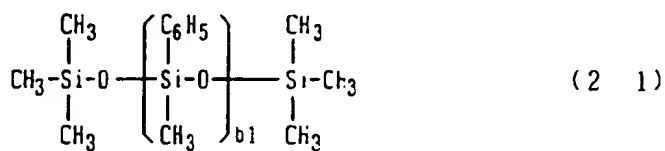
[0012]

(In the formula, a is a number between 3 and 20,000).

(B-2) The methyl phenyl polysiloxane represented by the following formula (2-1) or (2-2)

[0013]

[Structure 2]



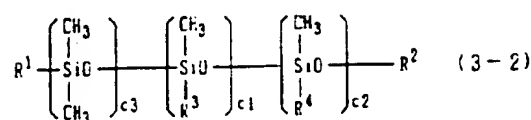
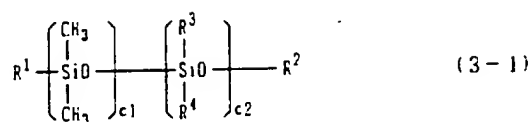
[0014]

(In the formulas, b1 represents a number of 1-20,000, and b2 and b3 represent numbers with their sum of 1-500).

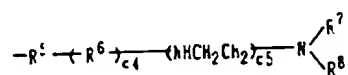
(B-3) The amino-modified silicone represented by the following formula (3-1) or (3-2)

[0015]

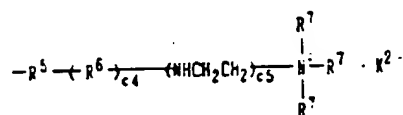
[Structure 3]



Here,  $R^1$  represents a methyl group or a hydroxyl group, and  $R^2$  represents a methyl group or a hydrogen atom.  $R^3$  represents an aminoalkyl group represented by



or



(where  $R^5$  is a divalent hydrocarbon group.  $R^6$  is a group of  $-OCH_2CH_2-$ ,  $\begin{array}{c} -OCHCH_2- \\ | \\ CH_3 \end{array}$ ,  $\begin{array}{c} -OCH_2CHCH_2- \\ | \\ OH \end{array}$ ,

or

$\begin{array}{c} -CCN_2CH- \\ | \\ CH_2OH \end{array}$ .  $R^7$  and  $R^8$  represent hydrogen atoms or monovalent hydrocarbon groups.  $c4$  and  $c5$  are integers of 0-6.  $X^{2-}$  represents a halogen ion or an organic anion).  $R^4$  is a hydroxyl group, a hydroxyalkyl group, an oxyalkylene group, a polyoxyalkylene group or a methyl group.  $c1$ ,  $c2$  and  $c3$  are integers depending upon the molecular weight.

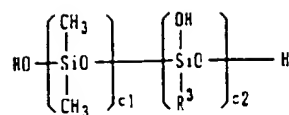
[0016]

Among these, the especially preferred amino-modified silicone is one represented by the following formula.



[0017]

[Structure 4]



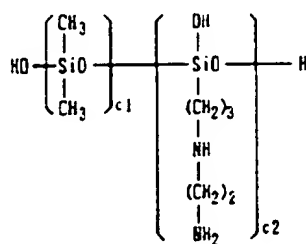
[0018]

(In the formula,  $\text{R}^3$ ,  $c1$  and  $c2$  have the meanings described previously.)

Furthermore, the typical amino-modified silicone is represented by the following general formula. Its average molecular weight is about 3000-100.000. This is described with the name of Amodimethicone in the CTFA Dictionary (U.S., Cosmetic Ingredient Dictionary) 3<sup>rd</sup> Edition.

[0019]

[Structure 5]



[0020]

(In the formula,  $c1$  and  $c2$  have the meanings described previously.)

It is preferable that the amino-modified silicone described previously be used as an aqueous emulsion. Said aqueous emulsion can be obtained by the emulsion polymerization of a cyclic diorganopolysiloxane and an organodialkoxysilane having an aminoalkyl group and a hydroxyl group, a hydroxyalkyl group, an oxyalkylene group, or a polyoxyalkylene group in the presence of a quaternary ammonium salt type surfactant and water, described in, for example, Japanese Kokoku Patent No. Sho 56[1981]-38609.

[0021]

Furthermore, if the amino-modified silicone is used as an aqueous emulsion, the amount of the amino-modified silicone contained in said aqueous emulsion is generally 20–60 wt%, preferably 30–50 wt%.

[0022]

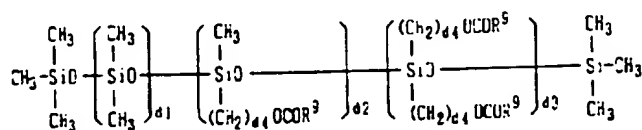
As commercial products of the preferred amino-modified silicone aqueous emulsions, SM 8702C (manufactured by Toray Silicone Co., Ltd.), DC 929 (manufactured by Dow Corning Co., Ltd.), and so on can be mentioned.

[0023]

(B-4) The fatty acid-modified polysiloxane represented by the formula (4) in the following

[0024]

[Structure 6]



[0025]

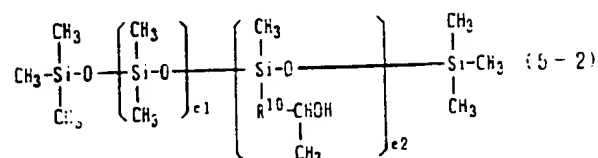
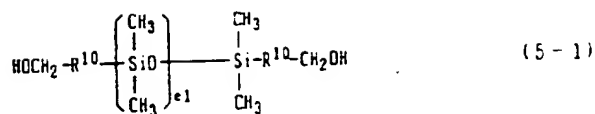
(In the formula, d1, d2, and d3 represent numbers of 1–350, and d4 represents a number of 0–10. R<sup>9</sup> represents C<sub>n1</sub>H<sub>2n1+1</sub> (where n1 = 9–21)).

(B-5) The alcohol-modified silicone represented by the following formula (5-1) or

(5-2)

[0026]

[Structure 7]



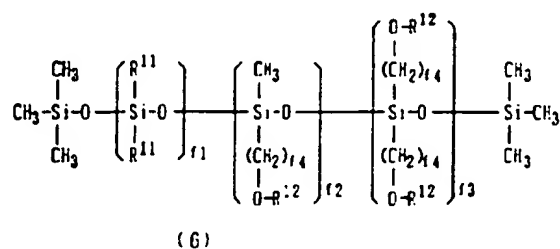
[0027]

(In the formula, e1 and e2 represent numbers of 1-500 (preferably 1-200), and R<sup>10</sup> represents C<sub>n2</sub>H<sub>2n2</sub> (where n2 = 0-4).

(B-6) The aliphatic alcohol-modified polysiloxane represented by the formula (6) in the following

[0028]

[Structure 8]



[0029]

(In the formula, R<sup>11</sup> is a methyl group or a phenyl group, f1 is an integer of 1-3000, f2 and f3 are integers such that f2 + f3 = 1-500, R<sup>12</sup> is a C<sub>1-28</sub> (preferably C<sub>12-22</sub>), and f4 is an integer of 0-6.).

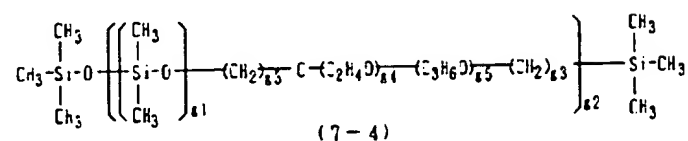
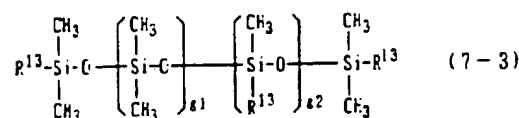
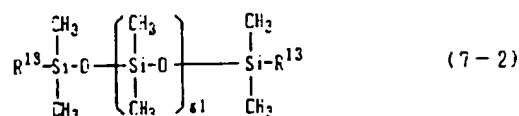
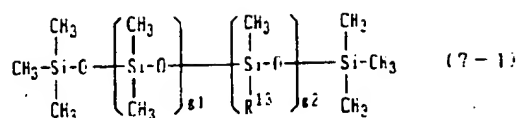
[0030]

(B-7) The polyether-modified silicone represented by formula (7-1), (7-2),

(7-3) or (7-4) in the following

[0031]

[Structure 9]



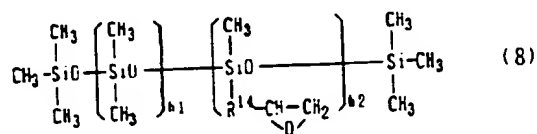
(Here,  $\text{R}^{13}$  is  $\text{R}^{13} = (\text{CH}_2)_{g3} - \text{O} - (\text{C}_2\text{H}_4\text{O})_{g4} - (\text{C}_3\text{H}_6\text{O})_{g5} - \text{B}$  (where B is a  $\text{C}_{1-12}$  alkyl group or a hydrogen atom),  $g1$  is 1-2000,  $g2$  is 1-1000,  $g3$  is 0-10,  $g4$  is 0-50,  $g5$  is 0-50 ( $g4 + g5 \geq 1$ )).

[0032]

(B-8) The epoxy-modified silicone represented by the following formula (8)

[0033]

[Structure 10]



[0034]

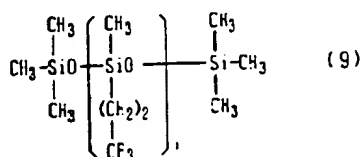
(In the formula, h1 is a number of 1-500 (preferably 1-250), h2 is a number of 1-50 (preferably 1-30), and R<sup>14</sup> is a C<sub>1-3</sub> alkylene group.)

[0035]

(B-9) The fluorine-modified silicone represented by the following formula (9)

[0036]

[Structure 11]



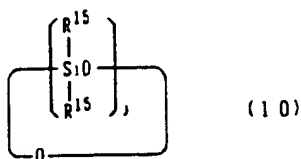
[0037]

(In the formula, i is a number of 1-400 (preferably 1-250).)

(B-10) The cyclic silicone represented by the following formula (10)

[0038]

[Structure 12]



[0039]

(In the formula, j is a number of 3-8, R<sup>15</sup> is a C<sub>1-3</sub> alkyl group.)

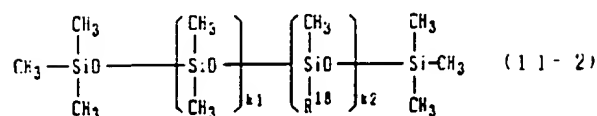
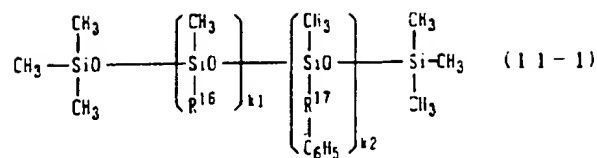
[0040]

(B-11) The alkyl-modified silicone represented by the following formula

(11-1) or (11-2)

[0041]

[Structure 3]



[0042]

(In the formula,  $k_1$  and  $k_2$  are numbers of 1–500 (preferably 1–200),  $\text{R}^{16}$  is a  $\text{C}_{2-18}$  alkyl group,  $\text{R}^{17}$  is  $\text{C}_{n3}\text{H}_{2n3}$  ( $n_3 = 0-4$ ),  $\text{R}^{18}$  is a  $\text{C}_{10-22}$  alkyl group.)

[0043]

Among these silicones, in the case of a rinse, a conditioner or other rinse-type hair cosmetic materials, those of (B-1) (In formula (1),  $a$  can be selected between 3 and 20,000 according to the objective of the finishing feel. However, it is preferably 100–1000 or so as a type of light finishing.), (B-3), (B-6), (B-7), and (B-10) mentioned previously are preferred. Furthermore, in the case of a hair cream, a styling lotion, a styling mousse or other nonrinsing hair cosmetic materials, those of (B-1) (In formula (1),  $a$  is preferably 2000–8000 for the objective of decreasing the oily feeling.), (B-2), (B-3), (B-7), and (B-10) mentioned previously are preferred.

[0044]

If the amount of blending of silicones of component (B) has a total of 0.01–30 wt%, especially 0.1–20 wt%, in the hair cosmetic material of the present invention, smoothness, softness, agent appearance, stability, and other aspects will be good. Silicones of component (B) can be used as such. The material of an emulsion type obtained by emulsification with a surfactant or the material obtained by emulsion polymerization may also be used.

[0045]

In the hair cosmetic material of the present invention, as components other than those mentioned previously, water, ethanol and other solvents, a surfactant, an oil, a polyvalent alcohol, a variety of pharmaceutically effective agents, a preservative, a perfume, and virtually all other ingredients appropriately used in ordinary hair cosmetic materials are appropriately selected and blended according to their objectives, applications, agent types, etc. For example, as the surfactants, linear or branched alkylbenzene sulfonates, alkyl or alkenyl ether sulfates with the addition of ethylene oxide and/or propylene oxide, olefin sulfonates, alkane sulfonates, saturated or unsaturated fatty acid salts, alkyl or alkenyl ether carbonates with the addition of ethylene oxide and/or propylene oxide,  $\alpha$ -sulfo fatty acid salt esters, amino acid type surfactants, phosphoric acid ester type surfactants, sulfosuccinic acid type surfactants, sulfonic acid type amphoteric surfactants, betaine type amphoteric surfactants, alkylamine oxides, linear and/or branched alkyl or alkenyl quaternary ammonium salts or other cationic surfactants, polyoxyalkylene alkyl or alkenyl ether, polyoxyalkylene alkyl phenyl ether, higher fatty acid alkanolamide or alkylene oxide adducts, esters of polyvalent alcohols and fatty acids, esters of sorbitol and fatty acids, esters of sucrose and fatty acids, esters of higher fatty acids and sugar, and so on can be used. The amount of blending of a surfactant into the hair-setting agent of the present invention is preferably 0–10%, especially preferably 0–5%. As the oils, stearic acid or other higher fatty acids, cetanol or other higher alcohols, cholesterol, Petrolatum, cholesteryl isostearate, sphingolipids or other solid fats, squalene, jojoba oil, as well as silicone derivatives, and other liquid fats and so on can be mentioned. As the polyvalent alcohols, glycerol, 1,3-butylene glycol, propylene glycol, dipropylene glycol, polyethylene glycol, polyglycerol, sorbitol, and so on can be mentioned. As solvents other than water and ethanol, benzyl alcohol, 2-benzyloxyethanol, N-alkylpyrrolidone, diethylene glycol monoethyl ether, and so on can be mentioned. As other ingredients, permanent agents, perfumes, coloring matters, ultraviolet light absorbers, antioxidants, trichlosane [transliteration], trichlorocarbene, and other sterilizers, glyceryl linic [transliteration] acid potassium salt, tocopherol acetate, and other anti-inflammatory agents, zinc pyrithione, Octopirox, and other anti-dandruff agents, methylparaben, butylparaben, and other preservatives, lactic acid, citric acid, and other pH adjusting agents, and so on can be added at will in a range without damaging the effectiveness of the present invention.

[0046]

The hair cosmetic material of the present invention can be prepared in a variety of agent types, that is, a spray, a mist, a gel, a lotion, a tonic, a blowing agent, a cream, a post-foaming gel, or the like, according to the conventional methods. Furthermore, in the case of an aerosol,

butane, isobutane, pentane, isopentane, and other volatile hydrocarbons, dichlorofluoromethane, dichlorotetrafluoroethane, and other halogenated hydrocarbons, dimethyl ether, carbon dioxide, nitrogen, air or other spraying gases are contained.

[0047]

#### Effect of the invention

The hair cosmetic material of the present invention prevents the adherence of scalp oils, other oily dirt, dust, and so on, is excellent in the style maintaining capability for hair in rain or at high humidity, and gives a smooth feel to hair, by maintaining water-repelling and oil-repelling characteristics.

[0048]

#### Application examples

The present invention will be explained more specifically with application examples in the following. However, the present invention is not to be restricted to these. The evaluations of the hair cosmetic materials were carried out in the following manner. Furthermore, "%" in the following represents "wt%."

[0049]

#### 1. Oil-repelling characteristics

The wetting power of the hair treated according to the present invention by n-hexadecane was evaluated by the Wilhelmi [transliteration] method. Healthy hair with an equal true circularity of at least 70% and without receiving a permanent hair dye or other chemical treatments was treated with the present agent. Its oil-repelling characteristics were evaluated from the angle of contact determined from the wetting power by n-hexadecane.

[0050]

Table I. Angle of contact: at least 60° ◎

Less than 60° to	O
Less than 50° to 40°	Δ
Less than 40°	X

[0051]

#### 2. Smoothness



By using the same hair as above, the dynamic frictional coefficient was evaluated by the nylon pulley method.

[0052]

Table II. Dynamic frictional coefficient: Less than 0.1 ◎

0.1 – 0.15	○
0.15 – 0.20	△
More than 0.20	X

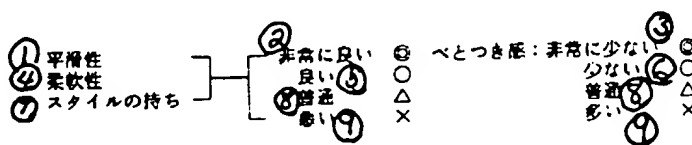
[0053]

### 3. Sensuous evaluation

With 10 panelists, treatments were carried out with the present agent and various comparative products. A sensuous evaluation was carried out on the smooth feeling, softness, stickiness, and style maintenance after one day.

[0054]

Table III



Key:

1	Smoothness
2	Very good
3	Stickiness: Very little
4	Softness
5	Good
6	Little
7	Style maintenance
8	Fair
9	Poor

[0055]

### Application Example 1

The hair rinse compositions shown in Table IV in the following were manufactured by the following manufacturing method and evaluated. The results are also shown in Table IV.

[0056]

Table IV

(A) 成分 (%)	(B) 本 発 明 品			(C) 比 較 品		
	1	2	3	1	2	3
(1) ステアリルトリメチル アンモニウムクロリド	2.0	2.0	2.0	2.0	2.0	2.0
(2) セチルアルコール	2.0	3.0	2.0	3.0	3.0	3.0
(3) ジメチルシリコン (10万cs)	1.0	5.0	0.1	—	1.0	—
(4) ポリテトラフルオロエ チレン微粒子 (粒子径0.01~1 μ. 平均径0.9 μ)	1.0	0.1	5.0	—	—	1.0
(5) 流動パラフィン	0.5	0.5	0.5	0.5	0.5	0.5
(6) ヒドロキシエチルセル ロース (1%水溶液粘 度8000cp)	0.3	0.3	0.3	0.3	0.3	0.3
(7) 香料	0.2	0.2	0.2	0.2	0.2	0.2
(8) 水	バランス	バランス	バランス	バランス	バランス	バランス
(9) 評価	1. 撥油性	○	○	○	×	×
	2. 平滑性	○	○	○	×	×
(10) 評価	3. 官能評価 (1日後)	○	○	○	×	×
	サラサラ感	○	○	○	×	×
	柔軟性	○	○	○	×	×
	べとつき感	○	○	○	×	×
	スチール持ち	△	△	△	×	×

- Key: A Components (%)  
 B Products of the present invention  
 C Comparative products  
 1 Stearyltrimethylammonium chloride  
 2 Cetyl alcohol  
 3 Dimethyl silicone (100,000 cs)  
 4 Polytetrafluoroethylene microparticles (particle diameters 0.01–1 μm, average diameter 0.9 μm)  
 5 Liquid paraffin  
 6 Hydroxyethylcellulose (1% aqueous solution, viscosity 8000 cp)  
 7 Perfume  
 8 Water  
 9 Balance  
 10 Evaluations  
 11 Oil-repelling characteristics  
 Smoothness

Sensuous evaluations  
 (after 1 day)  
 Smooth feeling  
 Softness  
 Stickiness  
 Style maintenance

[0057]

#### Manufacturing method

In (8) heated to 70°C, a mixture of (1), (2), (5), and (6) was added. After emulsification, it was cooled to 45°C while it was being stirred. (7) and a material obtained by good mixing of (3) and (4) beforehand were added. While it was being stirred, it was further cooled to room temperature to obtain a hair rinse composition.

[0058]

#### Application Example 2. Hair treatment composition

[0059]

Table V

(1) 2-Dodecylhexadecyltrimethylammonium chloride	1.5(%)
(2) Stearyltrimethylammonium chloride	2.0
(3) Cetostearyl alcohol	3.0
(4) Dimethyl silicone (1,000,000 cs)	2.0
(5) Tetrafluoroethylene/hexafluoro propylene copolymer microparticles (particle diameters 0.01–1 $\mu\text{m}$ , average particle diameter 0.7 $\mu\text{m}$ ).	2.0
(6) Liquid paraffin	3.0
(7) Hydroxyethylcellulose (1% aqueous solution viscosity 8000 cp)	0.5
(8) Polyoxyethylene oleyl ether (EO = 5)	0.5
(9) Methylparaben	0.2
(10) Perfume	0.4
(11) Water	Balance
Total	100.0

Application Example 4. Set lotion composition

[0063]

Table VII

(1) 2-Dodecylhexadecyltrimethylammonium chloride	0.5 (%)
(2) Silicone resin (softening temperature about 66°C) (Solid silicone resin P-1 described in Japanese Kokai Patent Application No. Hei 2[1990]-42008)	2.0
(3) Cyclic silicone (tetramer)	2.0
(4) Polytetrafluoroethylene microparticles (the material of Application Example 1)	2.0
(5) Liquid paraffin	0.5
(6) Methacrylic acid ester polymer (Yukaformer AM-75, manufactured by Mitsubishi Yuka Co., Ltd.)	1.0
(7) Polyethylene glycol	1.0
(8) Ethanol	20.0
(9) Perfume	0.3
(10) Water	Balance
Total	100.0

[0064]

The hair treated with the set lotion of the present invention had an excellent set-maintaining power at high humidity. Furthermore, stickiness due to sebum or the like was inhibited.

Application Example 5. Styling cream composition

[0065]

Table VIII

(1) 2-Dodecylhexadecyltrimethylammonium chloride	0.5 (%)
(2) Methyl phenyl silicone (b1 = 50 in formula (2-1))	1.0
(3) Polyether-modified silicone (g1 = 60, g2 = 5, g3 = 3, g4 = 15, g5 = 30, B = CH <sub>3</sub> in formula (7-1))	1.5
(4) Polyfluorotetraethylene microparticles (particle diameters 10-20 μm,	

average particle diameter 16 $\mu\text{m}$ )	2.0
(5) Acrylic acid ester-methacrylic acid ester copolymer (Plus Size L53P (manufactured by Gono Scientific Co.))	8.0
(6) Polyoxyethylene (10) hexadecyl ether	0.5
(7) Ethanol	5.0
(8) Perfume	0.2
(9) Coloring matter	0.01
(10) Water	Balance
(11) LPG (4.0 $\text{kg}/\text{cm}^2\text{G}$ , 20°C)	10.0
Total	100.0

[0066]

The hair treated with the styling cream of the present invention had an excellent set-maintaining power at high humidity. Furthermore, stickiness due to sebum or the like was inhibited.